Solid molybdenum nitride microdisc electrodes: Fabrication, characterisation, and application to the reduction of peroxodisulfate

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Abstract

A new methodology was developed to fabricate solid molybdenum nitride microdisc electrodes for the first time. The MoN microrods were produced by heating Mo microwires in dry NH3 atmosphere for several hours. They were characterised by scanning electron microscopy (SEM), energy dispersive spectroscopy (EDS) and X-ray diffraction (XRD). The latter revealed the samples had crystallised in the delta(3)-MoN phase with a core of gamma-Mo2N. Their electrochemical behaviour was probed for the reduction of Ru(NH3)(6)(3+). For this fast electron transfer the MoN microdisc electrodes returned similar voltammetric features to Pt microelectrodes. Their amperometric response was further tested with the reduction of peroxodisulfate. In contrast with other electrode materials, the reduction of S2O82- on MoN microdiscs delivered steady state voltammograms with well-defined diffusion controlled plateau. At low sweep rates, the limiting current was consistent with hemispherical diffusion and stable for at least 500 s. The diffusion coefficient of S2O82 derived from these results, 9.5 x 10 (6) cm(2) s (1), is in excellent agreement with previous work. At high sweep rates, the reduction of peroxodisulfate was found to be complicated by the simultaneous reduction of adsorbates. The results indicate that MoN is an ideal electrode material to monitor the concentration of peroxodisulfate under steady state conditions. (c) 2018 Elsevier Ltd. All rights reserved.

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